Magnesium Technology 2013
Edited by: Norbert Hort, Suveen N. Mathaudhu, Neale R. Neelameggham, and Martyn Alderman
TMS (The Minerals, Metals & Materials Society), 2013

ANNEALING OF COLD AND WARM ROLLED AZ31B MAGNESIUM ALLOY SHEETS

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Keywords: Magnesium, AZ31B, precipitation, $Mg_{17}Al_{12}$

Abstract

Annealing experiments have been performed on warm and cold rolled AZ31B magnesium alloy sheets at 200°C in several time intervals. The effect of the amount of deformation, the annealing time, and the presence of intermetallic phases were evaluated to obtain a microstructure constituted of fine recrystallized grains. Microstructural evolution was followed using optical microscopy, scanning electron microscopy combined with energy dispersive spectroscopy (SEM-EDS), X-ray diffraction and Vickers hardness measurements. After annealing for 60 min, it was found that static recrystallization mostly takes place on cold-rolled sheets with high thickness reduction, refining the grain size below 10 μm. Twins that were observed in warm rolled specimens nearly disappear and microstructure became suitable for further rolling process. Coarse phases of $Mg_{17}Al_{12}$ were found inside grains and fine precipitation of Al_4Mn and AlMn were detected at grain boundaries, which presented strong pinning effects to prevent the grain growth.

Introduction

Magnesium alloys have great potential as lightweight structural materials in automotive and electronics applications [1] because of their superior performance such as high strength, high strength-to-weight ratio, good thermal conductivity and shock-absorbing characteristics [2−3]. However, magnesium alloys show poor plasticity due to their limited slip´s systems (hcp structure) at room temperature [1–3]. Then in cold rolling process, plastic deformation takes place by basal slips and twinning. Meanwhile, at high rolling temperature, above 200 °C, non-basal plane slips, dynamic recrystallization and preferred growth of grain are activated [4]. In this sense, during warm rolling process, thermodynamically unstable microstructures containing defects, dislocations and sub-grains are produced, depending greatly of rolling temperature. On the other hands, the precipitate formation (second phase) and nodes of dislocation network also affect the final microstructure improving or getting worse. A lot volume of big precipitates are commonly associated with crack's formation during rolling process, but fine precipitates can offer pinning effects on grain boundary motilities and can promote dislocation slip on non-basal planes via cross-slip, moreover, they can generate a grain´s refinement and texture´s modifications.

It is known, that rolling process of Mg alloys at temperatures lower than 200°C can be improved by reduction of grain size because it increases ductility [5−6]. Consequently, the microstructure and mechanical responses of rolling process is potentially improved by annealing treatments in order to homogenized microstructure, minimizing the big size of second phase precipitates (manganese-rich particles), refining grain size, reducing segregation, increasing solid solubility, enhancing precipitate nucleation within the matrix, and generating a distribution of fine precipitates [7-8].

In this study, the effects of previous deformation before annealed treatment, the annealing time and the intermetallic phase´s formation during annealed of AZ31 rolled sheets are investigated in order to obtain a microstructure constituted of fine recrystallized grains, which will be enable to posterior rolling with high reduction.

Experimental details

Material

The material used in this research was the same sheet used in the study of effects of rolling temperature on the AZ31B magnesium alloy [10]. The nominal composition was approximately 3.34–3.63 wt% Al, 0.45–0.53 wt% Zn, 0.27–0.29 wt% Zn and balance Mg. At room temperature, two small strips (1 cm×5cm×2mm) were rolled in single pass and without any lubricant, one with 10% of thickness reduction and the other one with 15%. Larger rolling reduction would cause extensive side crack, then was chosen 250 ◦C as rolling temperature in order to reach 15% and 25% of thickness reduction. All samples were rolled, maintaining 3.4 m/min as a rolling speed. After rolling, small pieces of rolled samples were annealed at 200°C for different time intervals, beginning from 30 minutes and increasing to 240 minutes. Every annealed sample was cooled rapidly in cool water to avoid ($Mg_{17}Al_{12}$)-phase precipitation. Annealed responses were study measuring Vickers hardness with a 300g load and studying the microstructure evolution using optical and scanning electron microscopy (SEM). Metallographic preparations included polishing and etching surfaces with standard etchant (acetic picral). The lattice structure was determined from X-ray diffractogram (XRD) and precipitates were analyzed by scanning electron microscopy (SEM) equipped with an energy dispersive X-ray (EDX) spectrometer.

Result and discussions

Rolling processes

In previous studies, warm-rolled magnesium alloys have shown a moderate hardening due to precipitate's formations, which interfered on properties and microstructure of rolled samples depending on their sizes and distributions [10]. As a follow-up to that study, it was evaluated the effect of the intermetallic phases presence, amount of previous deformation, before annealing and annealing time. To begging, it is interesting to note that AZ31B samples showed similar hardening after cold and warm rolling deformation (figure 1). A thickness reduction of 10% increased the value hardness in 48%, this hardening was attributed to extensive formation of twinning and dense dislocation pile-ups within grains, especially, on bigger grains where twins were more prominent, resulting in reorientations and activations of basal planes slips. A thickness reduction over 15% could cause the fractures on AZ31B magnesium strips; hence 15% represents the upper limit of maximum deformation under compression at room temperature. In high rolling temperature (250 °C), samples with 10% and 25% of thickness reduction showed similar hardness values due to the interaction and competitions of different deformation mechanisms like recovery, dynamic recrystallization and formation of intermetallic phases. In figure 2, it was evidenced from microstructures of cold and warm rolled samples that plastic deformation strain generates large amounts of twinning, shear bands, microscopy voids and cracks inside precipitates, resulting in inhomogeneous deformations. The voids were formed preferentially at triple points and at those grain boundaries along which there was a high shear stress. In some research [9], these void formations were associated with grain boundary sliding and new grains formed by dynamic recrystallization during warm deformation.

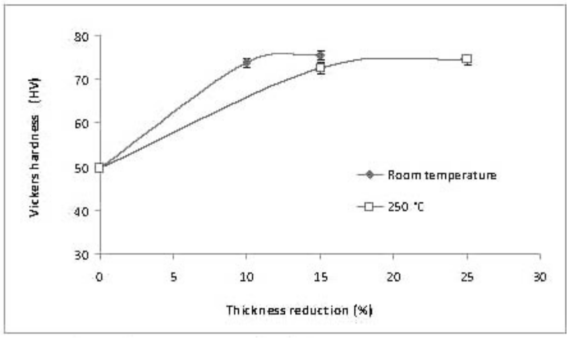


Figure 1 Microhardness values of rolled samples at room temperature and 250 °C.

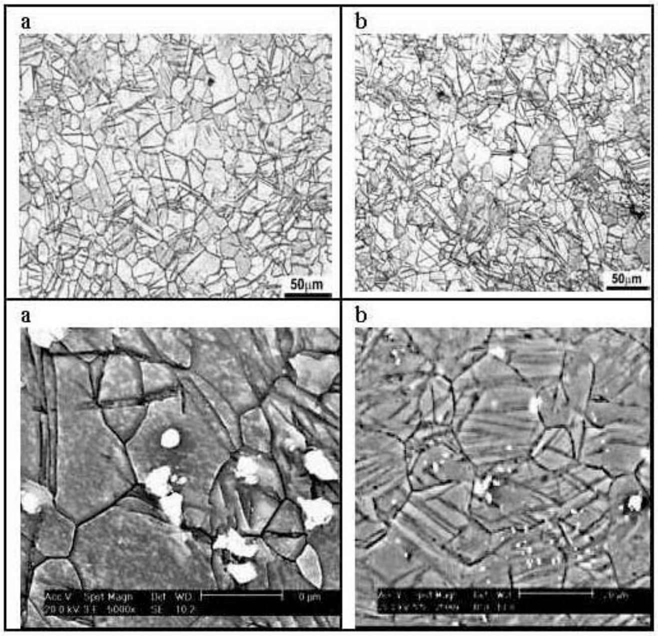


Figure 2 Micrographs of (a) cold and (b) warm rolled samples.

In the micrograph of cold rolled samples (figure 2a), it possible to see that grain size within the shear zones is somewhat smaller than in the regions without shear bands. Fine grains were formed by the higher strain rate in the shear zones, producing bimodal distributions of grain sizes with smaller grains about 5 μm and coarser ones about 25 μm. In warm rolling samples were also formed shear bands zones with fine recrystallized grains follow by precipitate formation that was not strongly affected by thickness reduction.

Annealing Treatment

The annealing times were showed remarkable effect on the twinning, the grain size's average and the precipitate's distribution. A period of 30 minutes of annealing treatment was not enough to disappear totally the twinning formed in rolling process (Figure 3-a1). Some big grains still showed traces of twinning; however, for a period of 60 minutes of annealing treatment, twins were growing or shrinking, leaving completely untwined grains. The stored energy has been lowered by static recovery and recrystallisation. The grain size within the shear bands was somewhat smaller than of the rest of grains due to the higher strain rate in the shear zones that produce finer structure in that regions.

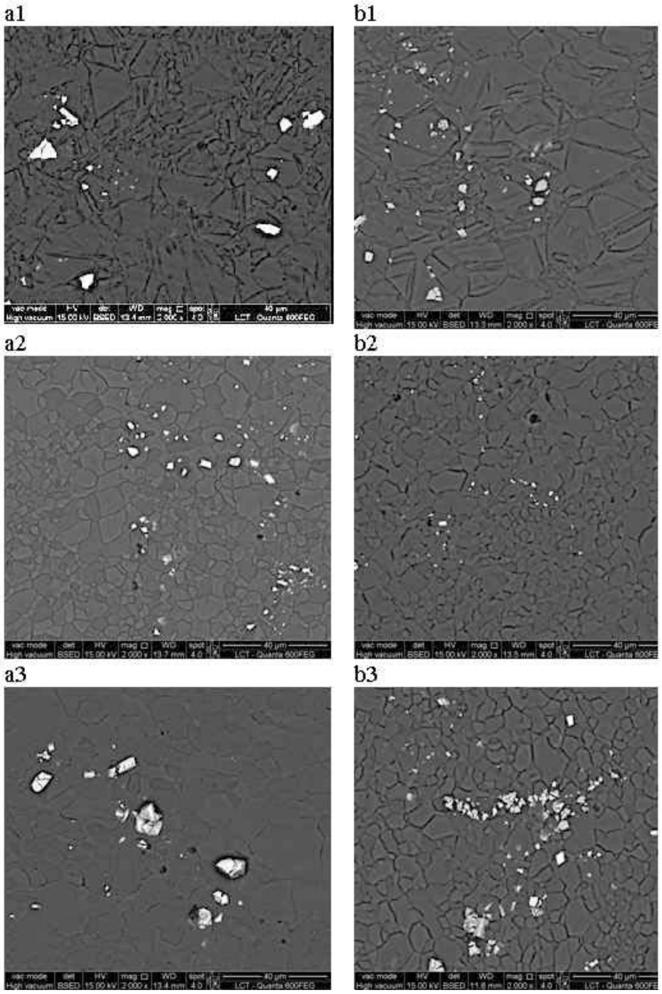


Figure 3 Microstructure of annealed specimens of cold (a) and warm (b) rolled samples (a1) 30 min annealed, (b1) 30 min annealed; (a2) 60 min annealed, (b2) 60 min annealed; (a3) 240 min annealed, (b3) 240 min annealed.

In micrographs of annealed samples (Figure 3), it was evidenced that 30 minutes of annealing treatments of cold rolled samples formed big-size precipitations which were distributed heterogeneously. However, the warm rolling applied prior to annealing increased the precipitate nucleation and formed fine precipitates. The thickness reductions (10% and 15%) affected slightly on hardness values of annealed samples, they showed different grain size and precipitate's distributions. In the Figure 4, it is evidenced that high thickness reductions significantly affects the hardening response due to precipitates formed during annealing treatment, especially after 2 hours of annealing. Since warm rolled samples contained small precipitates of $Mg_{17}Al_{12}$ and Al_4Mn, they were better redistributed after 60 minutes of annealing. In the Figure 3-c2, it possible to see a fine recrystallized grains structure with fine intermetallic particles. It is means that previous warm rolling significantly improved the precipitates redistribution after 1 hour of annealing treatments. Nevertheless, in longer annealing periods the size precipitates could increase a lot.

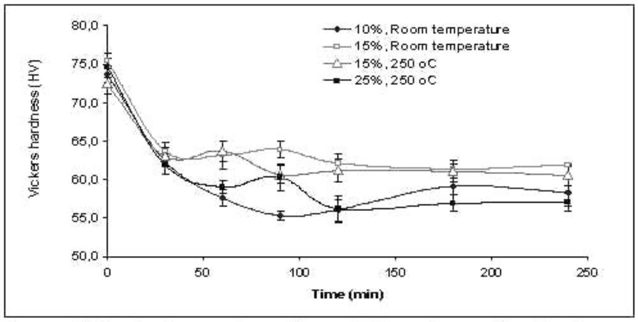


Figure 4 Microhardness values of annealed specimens of cold and warm rolled samples (at room temperature and 250 °C)

The hardening results of rolled and annealed samples suggest that intermetallic phases were partially solved and redistributed within matrix grains during warm rolling and annealing treatments, affecting on mobility of grain boundaries, recrystallization and precipitates redistribution.

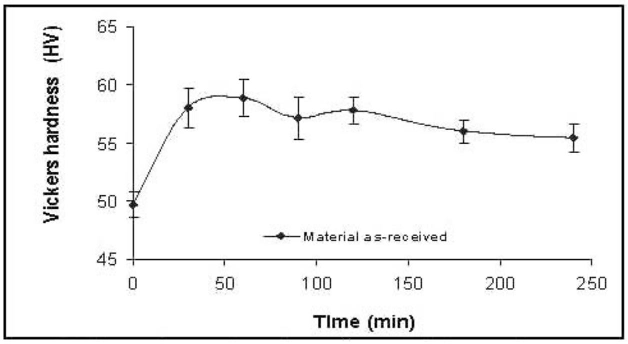


Figure 5 Microhardness values of annealing material as-received.

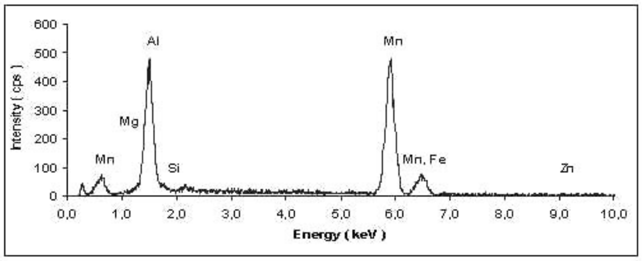
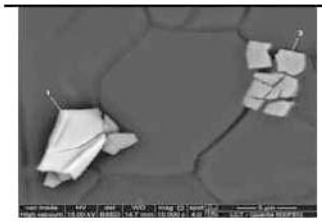


Figure 6 EDX precipitates spectrum at surface of warm rolled AZ31B-magnesium alloy.



Elem	Wt%	At%
MgK	2.36	3.88
AlK	31.39	46.47
SiK	2.16	3.08
MnK	61.67	44.84
FeK	2.42	1.73
Total	100.00	100.00

Figure 7 Precipitates micrographs and EDX spectrum result at surface of warm rolled AZ31B-magnesium alloy.

In the figure 7, it is shown precipitates on the grain boundaries and their SEM-EDX analysis showed that precipitates in AZ31B rolled samples contained roughly about 4 at.%Mg, 46 at.%Al and about 45 at.%Mn, which changed in composition in about 60at % Mg and 30 at% Al and about 9 at% Mn after annealed treatments. And, some fine particles may be formed by almost pure Al and Mn elements due to their limited solubility in magnesium (Figure 8).

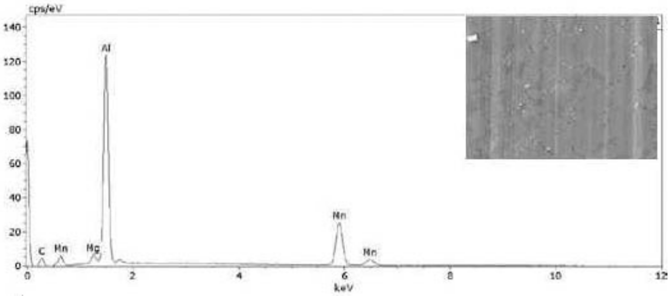


Figure 8 EDX precipitates spectrum at surface of annealed AZ31B-magnesium alloy.

The X-ray diffraction patterns of cold and warm rolled samples are shown in the figure 9. The intensity's differences of peaks were resulted by interaction of plastic deformation, dynamic recrystallization and transformation of precipitates into magnesium matrix.

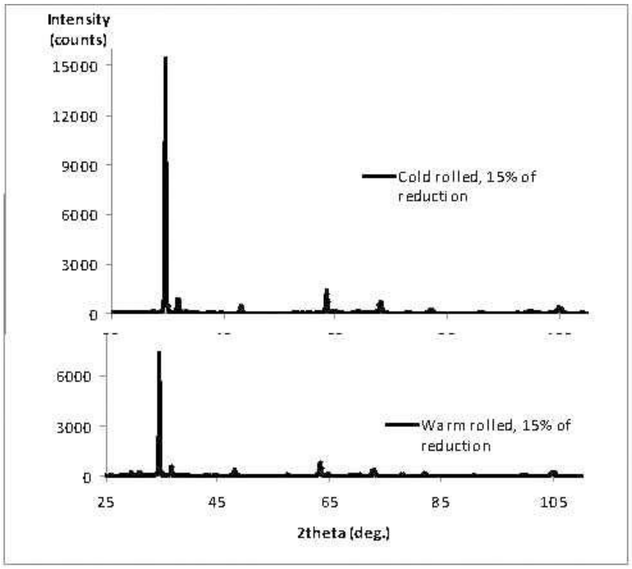


Figure 9 XRD diffraction patterns of AZ31B Mg alloy for rolled samples.

Comparing X-ray diffraction peaks of annealed samples with cold rolled sample and material as received (Figure 10), was evidenced that intensity of as-received material decreased by plastic deformation in cold rolling, resulting on broad peaks diffraction, meanwhile, after annealed, intensity of peak's diffraction increased due to volume fraction of precipitates grows, changing the lattice constants (Table 1). During rolling process and annealed treatments, the mechanism of interaction between plastic deformation and the formation of the eutectic precipitates ($Mg_{17}Al_{12}$, Al_4Mn and AlMn) for the AZ31 Mg alloys are still not totally clear. Meanwhile, it is possible to verify the presence of Mg_{17} Al_{12} and Al_4Mn precipitates with reference patters which coincided well with the diffractogram's peaks of rolled samples.

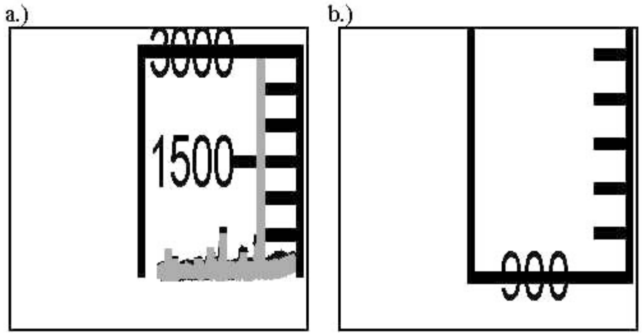


Figure 10 XRD diffraction patterns of (a) cold rolled sample and data peak's of $Mg_{17}Al_{12}$; (b) AZ31B Mg alloy for rolled and annealed samples

One way to analyze the phase´s diffusion is by calculating the lattice parameter "a" and "c". Noting the parameter values in the Table 1, interparticle spacing on the basal plane 'a' was greater than material as received and 'c' was smaller. And increasing the rolling temperature, this behavior is inverted, suggesting diffusion phenomena in the magnesium alloy. As on annealed samples, continuous precipitation was found to occur preferentially in the regions of the grains that were richer in manganese or aluminum, causing nonuniform precipitations. The regions of magnesium matrix that are higher in manganese or aluminum concentrations provided a higher driving force for precipitation.

Table 1 Values of lattice constants "a" and "c" determined by Rietveld method.

a) Samples cold and warm rolled of AZ31B magnesium alloy

Temperature (°C)	Thickness reduction (%)	a (nm)	c (nm)
	As received	3.198431	5.195763
25	10	3.202474	5.194697
25	15	3.204011	5.191890
250	15	3.204313	5.192003
250	25	3.201473	5.194611

b) Annealed samples at 200 °C for 4 hours

Temperature (°C)	Thickness reduction (%)	a (nm)	c (nm)
	As received	3.198930	5.195658
25	10	3.205553	5.193650
25	15	3.205378	5.193723
250	15	3.199044	5.196323
250	25	3.199793	5.196505

Conclusions

During cold rolling big precipitates were gradually broken by the large deformation and partly dissolved during warm rolling and annealing treatments, resulting in a redistribution of precipitates.

The recovery, recrytallization, grain growth and precipitation of second phases interacted in annealing treatments at 200°C, and the annealing microstructure change to homogeneous fine grains from mixed deformed big grains. If AZ31B Magnesium Alloys contain big precipitates with heterogeneous distribution, the grain sizes grow fast and the Vickers hardness decrease, especially to cold rolled samples with low thickness reduction. Since static recovery and recrystalization are develops on annealing process, the grain refinement could be achieved by 1 hours of annealing treatment of warm rolling.

Annealing treatments can improve the ductility by grains' refinements, by fine precipitate formation with homogeneous distribution.

The grain size and second-phase precipitates ($Mg_{17}Al_{12}$, Al_4Mn and AlMn) affect on mechanical properties, particularly hardness and their effects depends on level, size and precipitate's distribution, a high level of fine $Mg_{17}Al_{12}$ precipitates impeded extension twinning and enhanced dynamic recrystallization, resulting in a fine grain microstructure.

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